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# Effect of porcelain tile polishing residue on geopolymer cement

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ABSTRACT

The polishing of ceramic tiles surface produces high amounts of residual sludge, which requires deposition in controlled landfills. However, porcelain tile polishing residue (PPR) can be used as a partial source of aluminosilicate to produce geopolymer cement. Therefore, in this work, a geopolymeric cement based on PPR and metakaolin was developed using sodium hydroxide (NaOH) and sodium silicate ($Na_2SiO_3$) as activator solution. A $2^3$+1 factorial design was used with three factors: PPR content (15, 30 and 45 mass % in relation to metakaolin); NaOH (5, 6 and 7 mass %) and $Na_2SiO_3$ (48, 50 and 52 mass %). After synthesis, the samples were characterized by mechanical (compressive strength, hardness and dynamic modulus) and structural analyses (XRD and FTIR). The highest compressive strength was 72 MPa, obtained at 28 days (cured at 23 °C) in the composition containing 15% of PPR and 85% metakaolin, corresponding to a chemical composition with 53% $SiO_2$, 25% $Al_2O_3$ and 11% $Na_2O$, relation $SiO_2/Al_2O_3 = 3.44$, and a structure characterized as poly (sialate-disiloxo). The results showed that PPR can be used as an alternative source of raw material to produce geopolymer cement, while contributing to the reuse of an industrial waste.

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## 1. Introduction

The ceramic industry produces a large amount of waste. In China alone, more than one million tons of ceramic waste are generated each year (Sun et al., 2013). However, although this residue can be reused in the manufacture of some ceramic materials, most of it is deposited in controlled landfills (Rambaldi et al., 2007). Although research on the applications of porcelain tile polishing residue (PPR) in Portland cement materials is scarce (Wattanasiriwech et al., 2009; Andreola et al., 2010; Pelisser et al., 2012; Steiner et al., 2015), studies have shown the effects of the addition of PPR on Portland cement mortars and that partial replacement of Portland cement by PPR contributes to hydration. This effect was due to pozzolanic activity, filer effect and heterogeneous nucleation caused by the residue. The incorporation of PPR in the production of Portland cement is a well-established strategy for recycling this waste; however, this is true when a Portland cement factory is located in proximity to where the polishing residue is generated, and this does not always occur. Another application for this residue, considering its chemical composition, is in the production of geopolymeric cement, since it presents adequate concentrations of silica and alumina. Its use allows for the development of lower cost geopolymer cements with reduced environmental impact, due to the reuse of PPR.

The term “geopolymer” was devised by J. Davidovits in 1979 to describe the alkylation of silicates or aluminosilicates, forming inorganic polymers (Davidovits, 1991). Geopolymers consist of a three-dimensional network formed by several tetrahedral units of $SiO_4$ and $AlO_4$ connected with oxygen. They have amorphous to semicrystalline structures and their empirical formula can be described as: Mn $[-(SiO_2)_z-AlO_2]_n.wH_2O$, where z is 1, 2 or 3, M is the alkali metal cation and n is the degree of polycondensation (Davidovits, 1991). The International Union of Laboratories and Experts in Construction Materials, Systems and Structures (RILEM) considers geopolymers as a class of alkaline-activated materials and uses C-A-S-H (calcium aluminosilicate hydrate) and N-A-S-H (alkali aluminosilicate) acronyms to designate the hydration of the geopolymeric system that forms a gel structure, with high and low-calcium systems (Provis et al., 2015). Davidovits maintains hisoriginal statement of poly (sialate) structures, since hydrates do not

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form a polymer structure and thus, geopolymerization is not an alkaline-activated reaction, but an alkylation (Davidovits, 1991; Duxson et al., 2007). This remains a controversial issue.

Cementitious materials, such as geopolymers produced from fly ash, blast furnace slag or natural pozzolan, are competitive and they show low energy costs and low carbon dioxide emissions, reducing $CO_2$ emissions by 80% compared with Portland cement (Van Deventer et al., 2012). Some studies show the economic viability of the production of geopolymeric cements, despite the current limited demand for them. Some geopolymers can be obtained for US$ 150/m$^3$ (Ozel, 2012).

Sun et al. (2013) used another type of ceramic residue to obtain geopolymer cement and evaluated its mechanical behavior while considering the effect of temperature rise. After 28 days, the composition achieved a compressive strength of 71 MPa, which increased to 75 MPa after 2 h of exposure at 1000 °C. However, the ceramic residue was derived from municipal waste collection, a mixture of titles, pan forms, blocks and such, and was previously treated: the ceramics residue was first ultrasonically washed to remove contaminants such as paper scraps, metal, plastic, and organic matter, then the dried ceramics were crushed and pulverized in a ball mill for 45 min.

Given that PPR is a pozzolanic material, that its chemical composition is adequate for the production of geopolymeric cements, and the satisfactory results obtained by Sun et al. (2013), in this research, PPR from the ceramic tiling industry was evaluated in it capacity to produce geopolymers. The residue was used as a raw material to replace metakaolin and without prior treatment. The residue was collected from the base of the filter press and previously dried at a temperature of 60 °C, to control the humidity of the compositions evaluated. Preliminary studies showed homogeneity in the chemical and granulometric composition of this type of residue, which was collected once a week over a three-month period (Steiner et al., 2015). In this research, the optimal proportions of metakaolin and PPR were assessed, using the concentrations of the alkaline activators sodium silicate and sodium hydroxide as complementary variables. The results obtained enrich current research on geopolymers and show alternatives in the reuse of PPR as a binder in an economical and sustainable manner, while contributing to the development of special cements.

## 2. Materials and methods

### 2.1. Materials

The PPR was collected from a company specialized in surface polishing of ceramic tiles. Metakaolin was purchased from Metacaulim do Brasil. Sodium hydroxide (97% NaOH) and sodium silicate solution (27.5% $SiO_2$, 8.2% $Na_2O$ and 64.3% $H_2O$, mass %) were purchased from Sigma-Aldrich and Manchester. Table 1 shows the chemical composition (oxide mass %) of the metakaolin (MK) and PPR. PPR was collected in a filter press with a mean humidity of 30% and has a particle size $D_{50}$ of 13.7 μm. A more detailed characterization of the residue (XRD and particle size), collected once a week over a period of ten weeks, can be verified in the work of Steiner et al. (2015).

### 2.2. Sample preparation

A $2^3$+1 factorial design was used to elaborate the geopolymer compositions and the response surface methodology was used to analyze the results, following analysis of variance (ANOVA). The factors in the experiment were the degree of metakaolin substitution by PPR (mass %), and the concentrations of NaOH (mass %) and $Na_2SiO_3$ (mass %) (Table 2). The levels in the experimental design were: 15 and 45 mass % of MK substitution by PPR, with 30 mass % as central point; 5 and 7 mass % of NaOH addition, with 6 mass % as central point; and 48 and 52 mass % of $Na_2SiO_3$ solution, with 50 mass % as central point. The last column of Table 2 shows the amount of MK + PPR, the solid part of the mixture. The sum of NaOH with $Na_2SiO_3$ is the liquid part, the alkaline solution. In the notation of the compositions, first column, n was used for NaOH, and s was used for $Na_2SiO_3$. The first line shows the reference composition, without the substitution of MK by PPR. The last line shows the central point of the experimental design. The geopolymeric compositions were prepared according to the theoretical molar ration shown in Table 3.

The alkali solution (as per Table 2) was prepared by dissolving the NaOH flakes directly in the $Na_2SiO_3$ solution. Then the metakaolin and PPR were added to the reacting solution. The compounds were mixed in a mechanical blender for 4 min at 95 rpm. Next, the mixes were cast into plastic molds, sealed and set (cured) in a stove at 23 °C. The samples were stored in the stove until the time to execute the mechanical tests (7 and 28 days).

**Table 2**
Composition of the geopolymeric system. PPR is the polishing residue amount (mass %) substituting the metakaolin (MK), n is the NaOH amount (mass %) and s is the sodium silicate amount (mass %).

| Composition | PPR (%) | NaOH (n, %) | **$Na_2SiO_3$ (s, %)** | MK + PPR (%) |
|---|---|---|---|---|
| PPR0n2s2 (reference) | 0 | 6 | 50 | 44 |
| PPR15n1s1 | 15 | 5 | 48 | 47 |
| PPR15n1s3 | 15 | 5 | 52 | 43 |
| PPR15n3s1 | 15 | 7 | 48 | 45 |
| PPR15n3s3 | 15 | 7 | 52 | 41 |
| PPR45n1s1 | 45 | 5 | 48 | 47 |
| PPR45n1s3 | 45 | 5 | 52 | 43 |
| PPR45n3s1 | 45 | 7 | 48 | 45 |
| PPR45n3s3 | 45 | 7 | 52 | 41 |
| **PPR30n2s2** | 30 | 6 | 50 | 44 |

**Table 3**
Molar ratio of the as-prepared compositions of the geopolymeric system.

| Composition | $SiO_2/Na_2O$ | $SiO_2/Al_2O_3$ | $Na_2O/Al_2O_3$ | $Na_2SiO_3$/NaOH |
|---|---|---|---|---|
| PPR0n2s2 (reference) | 9.4 | 3.17 | 0.34 | 0.98 |
| PPR15n1s1 | 10.2 | 3.44 | 0.34 | 1.08 |
| PPR15n1s3 | 9.2 | 3.64 | 0.40 | 1.26 |
| PPR15n3s1 | 10.2 | 3.44 | 0.34 | 0.77 |
| PPR15n3s3 | 9.2 | 3.64 | 0.40 | 0.90 |
| PPR45n1s1 | 10.8 | 4.39 | 0.41 | 1.08 |
| PPR45n1s3 | 9.7 | 4.63 | 0.48 | 1.26 |
| PPR45n3s1 | 10.8 | 4.39 | 0.41 | 0.77 |
| PPR45n3s3 | 9.7 | 4.63 | 0.48 | 0.90 |
| PPR30n2s2 | 9.9 | 3.98 | 0.40 | 0.98 |

**Table 1**
Chemical analysis (oxide content) of the metakaolin and the PPR.

| Oxide (mass %) | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | $TiO_2$ | CaO | MgO | $K_2O$ | $Na_2O$ | $ZrO_2$ | LoI |
|---|---|---|---|---|---|---|---|---|---|---|
| Metakaolin | 54.0 | 42.0 | 0.5 | 0.5 | 0.1 | 0.1 | 0.5 | 0.1 | – | 2.0 |
| PPR | 67.7 | 21.0 | 1.8 | 0.7 | 2.6 | 1.1 | 2.3 | 1.8 | 0.3 | 0.8 |

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